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EVALUATION OF THE THERMAL RESISTANCE OF STRUCTURAL CERAMICS IN TESTING NOTCHED PRISMATIC SAMPLES

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The considered approach to determining thermal resistance in structural ceramics involves thermal cycling of samples with a special notch. The proposed thermal resistance characteristics include the loss of crack resistance after a thermal shock, the insensitivity of the material structure to defects formed at the notch apex as the result of a thermal shock, and the degree of defect accumulation at the notch apex. The testing results of ceramics made of Al_2O_3 , ZrO_2 , partly stabilized Y_2O_3 (molar part 3%), and cermet $\text{ZrO}_2 - \text{Y}_2\text{O}_3$ (molar part 3%) with metallic chromium additive (50 vol.%) are indicated.

In doing comparative testing of thermal resistance in structural ceramics, it is essential to use a method ensuring high reliability of the obtained results. This calls for maximum reproducibility of the results and a decrease in the spread of parameter values characterizing thermal shock resistance, within the limits of any considered sampling. A high degree of reliability also implies the possibility of extrapolation of test results from experimental prototypes to real large-scale products. At the same time, it is expedient to assess the thermal resistance of structural ceramics in the parameters which characterize the resistance of ceramics to the formation and evolution of thermal cracks.

It is known that the specified problems cannot be uniquely solved. This difficulty is largely due to the fact that structural ceramic, as a rule, represents a dense sintered body with a fine-crystalline structure, and, therefore, its destruction is controlled by small-sized structural defects commensurable with the crystal sizes. According to the classification in [1], these defects can be regarded as inhomogeneities (i.e., certain local areas in which dramatic alteration of density occurs) at the substructural level. The penetration of defects initiating destruction (known as critical defects (CD)) into the bulk of the material and their orientation with respect to the maximal stress direction is determined by the laws of mathematical statistics. This accounts, in particular, for the substantial spread in strength parameters within one series of samples produced by the same technology.

The widely used method of assessing thermal resistance based on the relative loss of strength in samples after a heat-

ing – cooling thermal cycle also yields a significant spread in the considered parameter values for the same reason. Furthermore, the critical defects in this case may be newly formed defects caused by the thermal shock, which differ in their size, geometrical shape, and orientation from one sample to another.

In our opinion, one can attempt to solve the above problem employing a new methodological approach, according to which a thermal shock is inflicted on a sample that contains a deliberately created CD of preset length, geometrical shape, and orientation. The following consideration should be taken into account. Such a CD is the sole stress concentrator in a ceramic structure, which controls its destruction under the effect of thermal or mechanical stress. Then the role of the statistical aspect related to the lack of certainty of a CD penetrating into the ceramic volume is to a large extent diminished. Consequently, it is possible to significantly decrease the spread in the measured parameter values. Furthermore, one can predict with a high degree of accuracy the thermal shock resistance of a real large-scale ceramic article, based on the test results of experimental samples with a deliberately created CD, since statistically (due to the large volume of such an article) the probability of the penetration of a random fracture-controlling CD, which in its extension and orientation is analogous to the one created in the experimental sample, is significantly higher. In that case, the fracture initiated under a thermal shock both in the experimental sample and in the real article will presumably occur under certain comparable values of critical stresses at the apex of the defect. Such an approach will probably help to overcome the role of what is known as the scale factor. Finally, in solving the latter problem, the use of a sample with a deliberately de-

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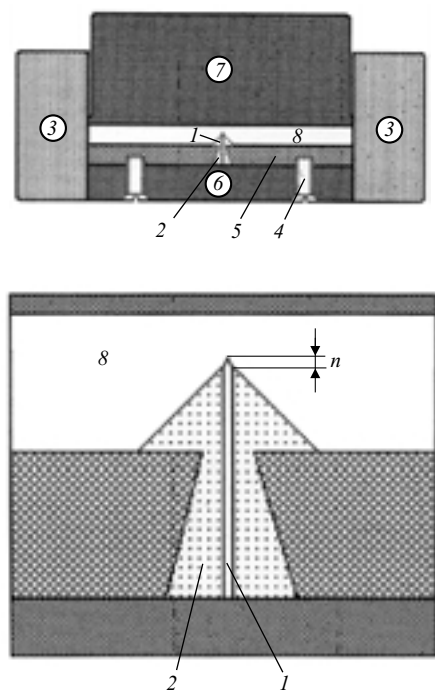


Fig. 1. Schematic diagram of the mold for molding notched samples: 1) steel blade; 2) wedge; 3) matrix; 4) fixing bolts joining parts 5 and 6 of the bottom punch; 7) upper punch; 8) sample; n) height of the blade sharpening.

veloped CD makes it possible in assessing thermal resistance to introduce certain parameters in the context of linear destruction mechanics.

A crack with a preset length C_0 can be used as a deliberately created CD. Such a crack, for instance, was caused by indentation in the surface layer of the sample [2], and the thermal resistance measure was the ratio $\Delta C/C_0$ (ΔC is the value of the crack increment after the thermal cycle). The disadvantage of this method is the impossibility of imparting a uniquely reproducible configuration to the developed crack from one sample to another. This is due to the fact that in the course of crack formation, the crack front interacts with the ceramics structure elements (grains, intergrain boundaries, micropores, inclusions, local stressed areas), which leads to irreproducible buckling of this front. The effect of indefinite configuration of the crack front can cause a great spread in the measured thermal resistance parameters. Furthermore, the known methods for producing cracks in ceramics are in most cases technologically complicated and labor-consuming [3].

To avoid the above shortcoming, we believe it advisable to use a special crack-modeling notch as the CD. However, a notch can adequately model a crack only when the curvature radius of its apex does not exceed a certain critical value ρ_{cr} . According to the known criteria [4, 5], $\rho_{cr} \leq T$ and $\rho_{cr} \leq 10d$ (T is the maximum opening of the crack in the material, and d is the average grain size). Assuming, by convention,

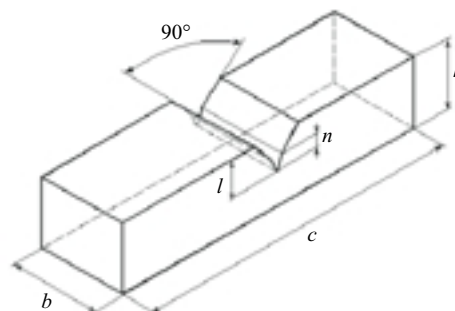


Fig. 2. Schematic drawing of the notched sample for thermal resistance testing: $l = 0.5h$; $n = 0.5$ mm; $b = 8$ mm, $h = 5$ mm, $c = 40$ mm.

$T = 10 \mu\text{m}$, $d = 1 \mu\text{m}$ and which is supposedly acceptable for dense fine-crystalline structural ceramics, we have $\rho_{cr} \leq 10 \mu\text{m}$ (*). In order to obtain reproducible thermal resistance characteristics in notched samples, it is essential to ensure the absence of accidentally induced defects in the notch apex area, which may be caused, for instance, by cutting the sintered material with a diamond tool, or the corrosive effect of gas or liquid media, or any other external effect. Otherwise, one should expect a substantial spread in the measured property values.

The present paper describes the method of studying thermal resistance in samples with a notch, which models a crack in dense ceramics, and discusses some testing results.

We suggested inflicting a thermal shock on prismatic samples in which a special notch had been made. The method for making the notch excludes the impact of a diamond tool on the sintered material. This makes it possible to satisfy the necessary condition: the absence of induced defects at the notch apex, which is required to ensure the reproducibility of the testing results. Such a notch was produced through molding in a specially designed mold (Fig. 1). Its specifics include the fixation of a steel blade 0.1 mm thick with a 14° sharpening angle into a wedge made of solidified epoxy resin with disperse corundum powder as the filler. The upper edge of the blade protrudes to the height of sharpening. The wedge surface is polished.

After the sample was taken out of the mold, its notch apex was identical to the configuration of the cutting blade edge (Fig. 2). After the end of shrinkage, the curvature radius of the notch apex in the sintered fine-crystalline samples was $5 - 8 \mu\text{m}$, which satisfies the conditions of adequate crack modeling (*). It should be noted as well that the described method of notch formation can be applied to a wide range of sintered materials.

The notched samples were tested for thermal resistance by two methods.

According to the first method, the thermal shock was inflicted by thermal cycling in the following mode: heating at a prescribed temperature – cooling in air at room temperature. For this purpose, the tested series of samples were placed on a ceramic mount, inserted in a preheated furnace, held for the

time required to equalize the temperatures of the sample and the furnace, and then taken out and left at the ambient temperature up to complete cooling.

According to the second method, a thermal-stressed state in the sample was developed by blowing the heated sample notch apex with a directed air jet having a temperature of 18°C (Fig. 3). In this case, the heat removal mainly occurred from the local volume at the notch apex. Such a thermal shock can be regarded as a local shock. To implement this method, the sample 1 was suspended on a nichrome wire 2 fixed on two corundum rods 3, which rely on a frame 4 made of porous corundum. The sample with the whole assembly was placed into a preheated furnace, heated to a prescribed temperature, and then removed from the furnace, after which a local thermal shock was inflicted. Owing to the sample being suspended, the heat removal from the local volume at the notch apex was implemented exclusively by the air flow. The air blowing was carried out from a metallic slot nozzle 5 (the nozzle slot cross-section was 0.5×10 mm) using a compressed air jet at a pressure of 0.5 MPa. The distance from the nozzle edge to the notch apex amounted to the half-height of the sample. The notch profile ensured free flow of air to its apex.

The following characteristics were proposed by us to evaluate the thermal resistance of structural ceramics:

$$\left(1 - \frac{K_{IC}^t}{K_{IC}}\right) \times 100\%; \quad (1)$$

$$\frac{\sigma_n^t}{\sigma}; \quad (2)$$

$$\frac{\sigma_n}{\sigma_n^t}, \quad (3)$$

where K_{IC} and K_{IC}^t are the critical stress intensity coefficient of samples before and after the thermal shock, respectively; σ is the bending strength of a smooth sample (without a notch); σ_n is the bending strength of a notched sample; σ_n^t is the bending strength of a notched sample after the thermal shock.

The mechanical testing of samples with and without notches was carried out according to the three-point loading scheme at a deformation rate of 1 mm/min. The procedure of calculating the specified parameters is described in detail in RF Patent No. 2131403. In the physical sense, ratio (1) is the value of relative crack resistance loss (percent) in structural ceramics after a thermal shock, and ratio (2) is the insensitivity (relative units) of structural ceramics to defects (i.e., to local microfractures) formed at the notch apex as a consequence of the thermal shock. The higher the value of ratio (2), the less sensitive to the specified defects the material is.

Ratio (3) characterizes the degree of accumulation (relative units) of the defects at the

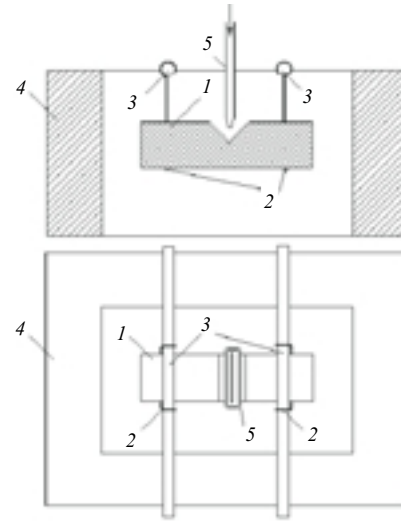


Fig. 3. Schematic diagram of the process of blowing the notch apex in a heated sample.

notch apex as a consequence of the thermal shock. This ratio can be found by dividing the ratio

$$\frac{\sigma_n}{\sigma} \quad (4)$$

by ratio (2). In this case, ratio (4) determines the insensitivity (rel. units) of the ceramic structure to the notch itself. Its value is inverse to the so-called effective coefficient of stress concentration [6]. Theoretically, this ratio cannot exceed 1 when measured within the thermoelastic range, and the value equal to 1 correlates with the complete insensitivity to the notch. The higher the value of ratio (3), the larger, presumably, the volume of the material occupied by local microfractures, which determines the decrease in the carrying capacity of the free cross-sectional area of the sample in front of the notch apex. Ratios (1) – (3) can be regarded as thermal resistance parameters that describe the stage of fracture initiation, when determined after the first thermal shock, and the stage of fracture evolution, when determined after a preset number of thermal cycles.

A metal ceramic material (cermet) based on ZrO_2 partly stabilized by Y_2O_3 (3 mol.%) with metallic chromium additives (50 vol.%) was developed for high-temperature service under the conditions of irregular heating and chilling. Its thermal resistance was assessed after one thermal cycle: 600°C – air (the first method). For reference purposes, ceramic samples without chromium additive were tested in the same mode of thermal cycling (Tables 1 and 2). It can be

TABLE 1

Material	Open porosity, %	σ , MPa	σ_n , MPa	σ_n^t , MPa	K_{IC} , MPa · m ^{1/2}	K_{IC}^t , MPa · m ^{1/2}
$ZrO_2 - Y_2O_3 - Cr$	5	286	116	108	3.7	3.6
$ZrO_2 - Y_2O_3$	2	693	167	151	6.0	5.4

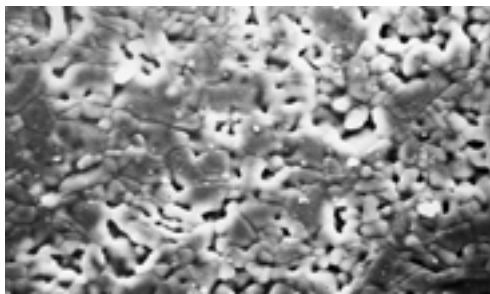


Fig. 4. Microstructure of reaction-fixed Al_2O_3 : polished section ($\times 867$).

seen that the heat-resistance parameters are better in cermet than in ceramics not containing metal. This is accounted for by the insignificant accumulation of local microfractures at the notch apex, owing to the fast elimination of the temperature gradient, due to the high thermal conductivity of the metal phase. In the case of ceramics without chromium additive, the local microfractures caused by irregular heating – cooling may to a large extent become supplemented by the defects initiated by the transition $t \rightarrow m$ of a certain quantity of grains under the thermal stress effect. Ratio (4) is higher for cermet than for ceramic without chromium additive, which is probably due to the possibility of a certain relaxation of applied stresses in the metal phase.

After a thermal shock, ratio (2) decreases, as compared to ratio (4), for both materials. This points to the decreasing insensitivity of their structure to the notch (which is a stress concentrator) after thermal cycling, due to the formation of defects at the notch apex, which become new stress concentrators. Their quantity, size, shape, distribution density, and orientation with respect to the notch apex determine the values of ratios (1) and (3). Apparently, the longest and the most rectilinear microcracks, whose orientation coincides with the notch direction, contribute to the most perceptible

TABLE 2

Material	$\frac{\sigma_n}{\sigma}$, rel. units	$\frac{\sigma_n^t}{\sigma}$, rel. units	$\frac{\sigma_n}{\sigma_n^t}$, rel. units	$\left(1 - \frac{K_{IC}^t}{K_{IC}}\right) \times 100$, %
$\text{ZrO}_2 - \text{Y}_2\text{O}_3 - \text{Cr}$	0.41	0.38	1.07	3
$\text{ZrO}_2 - \text{Y}_2\text{O}_3$	0.24	0.22	1.10	10

TABLE 3

Aluminum oxide material	Open porosity, %	σ_n , MPa	σ_n^t , MPa	$\frac{\sigma_n}{\sigma_n^t}$, rel. units	K_{IC} , $\text{MPa} \cdot \text{m}^{1/2}$	K_{IC}^t , $\text{MPa} \cdot \text{m}^{1/2}$	$\left(1 - \frac{K_{IC}^t}{K_{IC}}\right) \times 100$, %
Sintered	0	104	113	0.92	3.7	4.0	– 8
Reaction-fixed	2	116	146	0.79	4.2	5.1	– 21

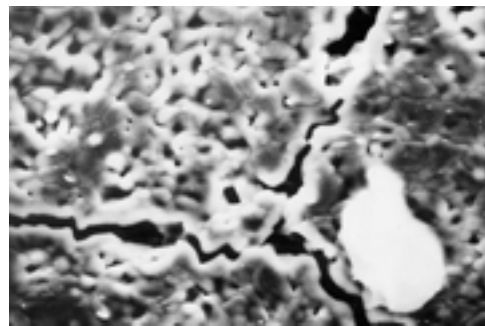


Fig. 5. Thermal cracks obtained on a polished surface of reaction-fixed Al_2O_3 after 850°C – water thermal cycle ($\times 867$).

decrease in the carrying capacity of the free cross-sectional area after the thermal shock.

If ratios (1) and (3) after the first thermal shock are found to be sufficiently high, one can draw an unambiguous conclusion of the unsuitability of using the tested material as a structural one. It can also be assumed that the proposed parameters (1) and (3) in this case are the quantitative characteristics of the initial stage of destruction related to crack initiation after a single thermal shock. In this case, of the two materials considered, this stage is more dangerous for the ceramic without chromium additive and less dangerous for metal ceramic.

A local thermal shock: 850°C – air (the second method) was used to evaluate the thermal resistance of two types of aluminum oxide materials: the first one sintered from finely dispersed Al_2O_3 powder + MgO (0.5 wt.%), i.e., powder A, and the other produced by reaction bonding and subsequent sintering of a batch containing a mixture of powder A and fine aluminum powder (35 vol.%), to provide for the exothermic reaction of aluminum oxidation. It was found (Table 3) that no decrease in strength and crack resistance is registered in both types of materials after a local thermal shock. Moreover, one can even talk about a certain probable tendency for increasing these parameters. Their increment value for the reaction-bonded Al_2O_3 exceeds the measurement error (7 – 10%).

All values of mechanical properties in the latter type of corundum are higher than in sintered Al_2O_3 . This is presumably due to the reinforcing effect of the secondary aluminum oxide phase, which is formed in oxidation of aluminum and envelops the initial Al_2O_3 grains (the primary aluminum oxide phase). Moreover, its fine-pore structure (Fig. 4), which is apparently formed as a consequence of zonal sintering [7, 8] of highly dispersed aluminum oxidation products, ensures resistance to the development of thermal cracks by means of multiple alteration of the direction of the crack-front expansion and the increase in the front extension, as it intersects the system of pores (Fig. 5). Such mechanism

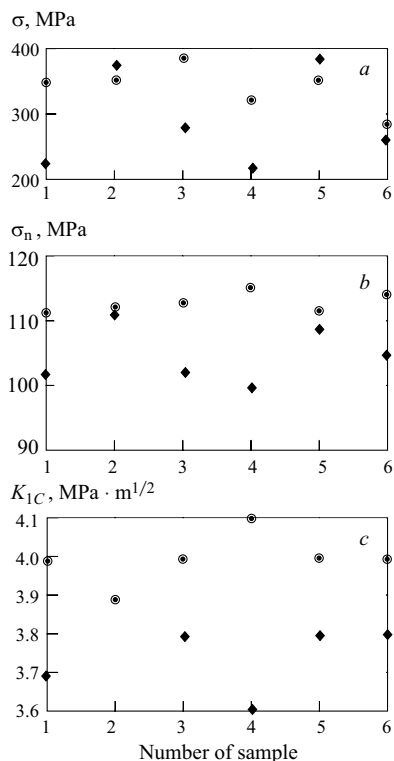


Fig. 6. The spread in the measured values of mechanical properties of samples (sintered Al_2O_3) without (a) and with a notch (b, c): ♦) before the thermal cycle; ○) after the thermal cycle.

of fracture retarding was successfully implemented, for example, in ceramics with the laminar-granular structure [3, 9], which has high thermal resistance.

Ratio (3) in both materials is less than 1. This means that no accumulation of defects at the notch apex has occurred as a result of the thermal shock. On the contrary, one can assume that a certain "healing mechanism" for structural defects in ceramics exists under certain conditions of thermal loading [10]. The negative values of parameter (1) point to the increased crack resistance of the samples as a consequence of the thermal shock, which does not contradict the modification in ratio (3) registered in this testing.

It should be noted that the above tendency toward the improvement of the mechanical properties of aluminum oxide samples was not revealed in the traditional method of evaluating thermal resistance based on the value of the relative change in strength (percent) after the thermal cycle (850°C – air) in samples without a notch, within the limits of the standard sampling (6 – 10 samples). This tendency could be revealed only after testing a fairly representative sampling (21 samples) and statistical processing of the results obtained [10]. This fact is related to the significant spread in the measured values of σ due to the brittle fracture in ceramics (Fig. 6a). For this reason, the σ increment value could not be

registered after the thermal shock on the background of regular fluctuations in this parameter value.

In the case of testing notched samples (within the limits of the same sampling), the spread in the parameter values σ_n and K_{1C} is significantly lower (Fig. 6b and c). This is due to the unique reproducibility of the configuration and the curvature radius of the notch apex, as well as the notch orientation with respect to the applied load vector. The notch is the deliberately developed concentrator of stresses acting in a local area at its apex under thermal and mechanical loading. Owing to this, a standard-size sampling of test samples with a special notch makes it possible to register the strength variation tendency, which would not be identified through testing the same quantity of samples without a notch.

It should be noted that the insignificant spread in the values of the σ_n and K_{1C} parameters measured before and after the thermal shock makes them more structure-sensitive, compared with the parameters σ obtained before and after the thermal stress effect. This means that by using these parameters, one can register even a slight strength variation, which can be caused by introducing special techniques (for instance, introduction of doping additives, formation of multicomponent powder mixtures, use of powders with a prescribed geometrical shape of particles, modification of time/temperature heat-treatment parameters) in the course of developing special ceramic structures.

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